PCT/AU2004/001389

AUSTRALIA

Patent Office
Canberra

I, JULIE BILLINGSLEY, TEAM LEADER EXAMINATION SUPPORT AND SALES hereby certify that annexed is a true copy of the Provisional specification in connection with Application No. 2003905590 for a patent by CYCLOPLAS HOLDINGS PTY LTD as filed on 13 October 2003.

COMMONWEALTH OF AUSTRALIA
AUSTRALIA
PATENT OFFICE

WITNESS my hand this
Twentieth day of October 2004

J. Billingsley

JULIE BILLINGSLEY
TEAM LEADER EXAMINATION
SUPPORT AND SALES

AUSTRALIA

Patents Act 1990

Cycloplas Holdings Pty Ltd

PROVISIONAL SPECIFICATION

Invention Title:

Process for Recycling Waste Plastics

The invention is described in the following statement:

Technical Field

The present invention relates to a process and apparatus for the recycling of waste plastics such as are found in municipal solids waste. The invention also relates to production of a wide range of value added items manufactured by the process.

Background Art

Conventional plastic recycling processes aimed at reuse of used plastic items have required pre-sorting into defined plastic types, followed in most cases by segregation of the plastic from contaminants such as coloured lids and labels and washing of the plastic to remove soil and other forms of latent matter. Following such preparation, the sorted washed plastics are granulated and re-used either in combination with virgin plastics of the same type or in combination with defined groupings of plastic requiring specified percentages of the sorted plastics to produce reliable and re-useable products.

However, these existing plastics recycling processes have several disadvantages. Whilst it has been found that between 10 - 25% of sorted, washed recycled material can be incorporated into new products, the quality of the resulting plastic products limits their use due to the inclusion of such reclaimed material. This is the case, for example, when PVC pipe is extruded for the carriage of potable water, only reground virgin PVC, but not recycled PVC, can be used. Furthermore, considerable costs are involved in preparing used plastic items for re-use, including the cost associated with the pre-sorting, segregation, washing and granulation of the recycled plastic prior to its conversion into reusable items.

Extensive source separation of plastic items is commonplace in modern society, which greatly facilitates the recycling of some commercially valuable plastics species such as PET and HDPE. When these are withdrawn from the waste stream, a large fraction of other plastics remains, which are of substantially lower commercial value and are usually disposed to landfill. Alternatively, the waste can be converted to energy by such processes as gasification. Concerned parties see both fates as ecologically unacceptable.

While it is expected that traditional processing will continue to be utilised for the recycling of the higher value plastics, the present invention enables the conversion of the large fraction of plastic remaining in the waste stream into useable product.

Therefore, the present invention presents an unexpected enhancement of the methods and technologies available for the recycling of all plastics. This process for the recycling of mixed waste plastics employs techniques and apparatus, which have not previously been possible for this application, thereby offering an environmentally acceptable purpose and reducing the cost of processing these materials into reusable products in an efficient manner.

Previously, the recycling of unsorted mixed plastic material has been found to be problematic as the materials in the mixture have different processing and melting characteristics. In order to over come this problem in the past, the plastics have been sorted based on common chemical characteristics which is time consuming and expensive. The sorted plastics are then heated to melting and used to form new articles.

The present inventors have developed an alternative process to recycle used plastics, particularly mixed plastic waste.

Disclosure of Invention

In a general aspect, the present invention relates to a process for recycling used plastic material by providing a desired dielectric property to the plastic and melting the plastic using microwave energy.

In a first aspect, the present invention provides a process for recycling plastic material comprising:

(a) reducing a sample of plastic material to form plastic particles having a desired particle size;

(b) treating the plastic particles with an agent which imparts a defined dielectric property to the plastic particles; and

(c) treating the treated plastic particles with microwave energy to form a molten plastic material.

The process may further comprise:

(d) forming the molten plastic material into a solid product.

Preferably, the agent which imparts a defined and increased dielectric property to the plastic particles is a microwave susceptor agent which provides an increased dielectric property to the plastic particles.

Preferably, the process further comprises providing a bonding agent to the plastic particles which assists in forming a cohesive molten plastic material..

In a preferred form, the microwave susceptor agent is provided with the bonding agent.

The process according to the present invention is particularly suitable for the treatment of mixed or unsorted plastics.

Preferably, the plastic particles are formed by shredding or grinding the mixed plastic waste. Other suitable methods can be used to reduce the plastic material to a desired particle size such as commercially available plastic granulation, shredding or pulverisation equipment.

The particle size can be less than about 50 mm. Preferably, the particle size is about 0.5 – 20 mm. More preferably, the particle size is between about 1 – 5 mm. It will be appreciated, however, that the particle size can vary depending on the waste plastic material and the agents used.

Preferably, the microwave susceptor agent has a dielectric constant higher than that of the plastic particles. In a preferred form, the microwave susceptor agent is also a colouring agent. Preferably, the agent is carbon black. In a preferred form, the bonding agent contains the microwave susceptor agent which is receptive to microwave energy and heats up preferentially under microwave treatment. It will be appreciated that other colouring agents that are compatible in plastic may also be used together with other materials that are microwave susceptors or enhancers which would also be suitable for the present invention. Examples of other such suitable microwave susceptor agents include, but are not limited to, those shown in Table 1:

TABLE 1

Microwave susceptor agent
HYDROCYANIC ACID
HYDROGEN PEROXIDE
TITANIUM DIOXIDE
TRIMETHYLSULFANILIC ACID
HYDROGEN FLUORIDE
FORMAMIDE
GLYCERIN, LIQUID
ACETAMIDE
FORMIC ACID
METHYL ALCOHOL
p-NITRO ANALINE
DIMETHYL SULFATE
HYDRAZINE
MALEIC ANHYDRIDE
TITANIUM OXIDE

The bonding agent, if used, is preferably added in proportions of between 1% to 15% (w/w).

Preferably, the bonding agent is a resin formed by dissolving soluble plastics used commonly in the art in industrial solvents well known in the art. The solvent used may also be from a recycled source. Soluble plastics, such as polystyrene (PS), and solvents such as thinners, toluene or acetone have been found by the present inventors to be particularly useful. Other soluble plastics and solvents, well known to the art, would also be suitable for preparation of the bonding agent. Examples include, but not limited to, those shown in Table 2:-

TABLE 2

Solvent	Plastic
m- Chlorobenzene	AC
Cyclohexane	AC, Nylon, PEEK, PS
Cyclohexanone	PVC, PS
Ethyl chloride	PP
Ethyl ether	PP
Furfuryl alcohol	PVC, HDPE
Isopropyl ether	AC
Ketones	AC
Methyl acetate	PP
Methyl chloride	PP, PVC
Methyl ethyl ketone	PVC, PPMA
Methylene chloride	PEEK
n-Octane	PP
n-Pentane	PP
Tetrahydrofuran	PEEK
Trichloroethylene	AC
Triethanol Amine	AC

In a preferred form, the plastic particles, the bonding agent containing the microwave susceptor agent and colouring are placed in a vessel equipped with a slowly rotating propeller where the elements are evenly combined to form a coated plastic material Other vessels in which the coated plastic material can be blended by stirring, tumbling, or other similar processes are also suitable for the present invention.

In a preferred form, a vacuum is applied to the coated plastic material to remove any harmful vapours generated during the heating stage of the process.

Preferably, the vacuum is maintained between 60 and 260 milliBar absolute pressure.

In a preferred form, the contents of the vessel are heated with microwave energy to a temperature of at least about 150°C but not exceeding about 230°C.

The microwave frequency used in the present invention may be of a frequency in the order of 915 mHz or 2.45 gHz. These frequencies are those permitted for use in industrial microwave applications in Australia but other frequencies may also be used in the present invention. One advantage of the use of microwave technology is that the heat generated by the interaction of the applied microwave energy with the microwave susceptor agent added in this process is uniformly distributed throughout the coated waste plastic and therefore plastics which are normally quite insulatory and difficult to heat by traditional means are efficiently and uniformly heated.

It will be appreciated that the amount of energy required will depend on the quantity of coated waste plastic being treated at any particular time and the type and ratio of microwave susceptor agent used.

In a preferred form, the microwave heating is applied without agitation, for a period not exceeding about 10 minutes and the heat is then continued with agitation. In another preferred form, the heating period used in the present invention is between 10 – 200 minutes. More preferably, the coated plastic material is heated less than about 150 minutes. Preferably, the contacting period is less than about 50 minutes. It will be appreciated that the duration of heating can vary depending on the volume of coated plastic material and the heating apparatus used.

Once molten, the plastic material can be conveyed while still hot to any suitable moulding equipment where it can be compression moulded, injection moulded or extruded in conventional plastic forming equipment.

The process according to the present invention may be performed batch-wise or continuously in a suitable vessel or through a suitable apparatus such as a screw conveyor. When a batch method is used, the components can be loaded and unloaded into a treatment vessel manually. When a continuous flow mode is desired, commercially available materials-handling equipment utilising a conveyor to feed the final mixture through the heating stage can be used.

In a second aspect, the present invention provides recycled mixed plastic material obtained by the process according to the first aspect of the present invention.

The recycled material according to the present invention is suitable for, but not limited to, the formation of a wide range of concrete and timber replacement products. Examples are shown in Table 3:

TABLE 3

Industry	**Product**
Agricultural	Fence posts Stakes
Building & Construction	Grates Bases for temporary fencing Head walls Hydrant Surrounds Man Holes & Covers Pipe – stormwater - sewerage - irrigation
Garden Furniture & Horticulture	Planter Boxes Decorative Sleepers Nursery Pots Garden Seating & Tables
Industrial	Pallets Bins
Landscaping	Grills Pavers Decking
Recreation	Wharf timbers Decking
Traffic Control	Bollards Parking Buffers Kerbs & Gutters

Throughout this specification, unless the context requires otherwise, the word "comprise", or variations such as "comprises" or "comprising", will be understood to imply the inclusion of a stated element, integer or step, or group of elements, integers or steps, but not the exclusion of any other element, integer or step, or group of elements, integers or steps.

Any discussion of documents, acts, materials, devices, articles or the like which has been included in the present specification is solely for the purpose of providing a context for the present invention. It is not to be taken as an admission that any or all of these matters form part of the prior art base or were common general knowledge in the field relevant to the present invention as it existed in Australia before the present invention was made.

In order that the present invention may be more clearly understood, preferred forms will be described with reference to the following drawings and examples.

Brief Description of the Drawings

Figure 1 shows a schematic diagram of apparatus suitable for the process according to the present invention.

Mode(s) for Carrying Out the Invention

Waste Material

Plastic materials that can be recycled by the process and apparatus of the present invention include, but are not limited to the plastic categories set out in Table 4.

TABLE 4

Acronym	**Chemical or Common Name**
AC	Acetal
ABS	Acrylonitrile butadiene styrene
ASA	Acrylate styrene acrylonitrile
Acronym	**Chemical or Common Name**
CA	Cellulose acetate
EVA	Ethylene vinyl acetate

EVOH	Ethylene vinyl alcohol
HDPE (PE-HD)	High density polyethylene
HIPS	High impact polystyrene
LDPE (PE-LD)	Low density polyethylene
LLDPE (PE-LLD)	Linear low density polyethylene
MDPE (PE-MD)	Medium density polyethylene
PA6	Polyamide (Nylon) type 6
PA11	Polyamide (Nylon) type 11
PA12	Polyamide (Nylon) type 12
PA66	Polyamide (Nylon) type 66
PBT	Polybutylene terephthalate
PC	Polycarbonate
PEEK	Polyether ether ketone
PE	Polyethylene
PES	Poly ether sulphone
PMMA	Polymethyl methacrylate (Acrylic)
POM	Polyoxymethylene (Acetal, Polyformaldehyde)
PPO	Polyphenylene oxide
PP (PPN)	Polypropylene
PPC	Polypropylene copolymer
PPH	Polypropylene homopolymer
PS	Polystyrene
PTFE	Polytetrafluoroethylene

Acronym	Chemical or Common Name
PVC	Polyvinyl chloride
PVC-C	Chlorinated polyvinyl chloride
PVC-P	Plasticised polyvinyl chloride
PVC-U (uPVC)	Unplasticised polyvinyl chloride
SAN	Styrene acrylonitrile copolymer
TPE	Thermoplastic elastomer
TPO	Thermoplastic olefin
TPU	Thermoplastic polyurethane
TPR	Thermoplastic rubber
XPS	Expandable polystyrene

APPARATUS

An apparatus for performing the process according to this invention in batch mode is shown diagrammatically in Figure 1.

The apparatus comprises the following components

Vessel (1) equipped with a stationary lid having an O-ring seal and means for microwave entrapment. The vessel is held closed by the applied force of an applied vacuum. When vacuum is released, the vessel is lowered providing unimpeded access to add or remove material for treatment.

Stirrer & Motor (2) with reduction gearbox and speed control to provide variable mixing speed and high torque required to mix the molten plastic by the action of a propeller mixer.

Thermocouple (3) to sense the temperature of the melting material in the vessel. The thermocouple, which is a standard Type K, is mounted so that it is earthed to the wall of the vessel and does not act as an aerial for microwaves.

Data Logger & Temperature Control (4) to record heating profiles and control the microwave energy delivery by the microwave generator to the material being heated in the vessel

Control Data (5) to switch microwave generation by the microwave generator (6).

Microwave Generator (6) to provide heating energy to the vessel (1) equipped with a 1 kW, 2.45 gHz magnetron and power supply.

Waveguide & Window (7) to conduct the microwave power from the microwave generator (6) to the vessel (1) to heat the material being processed. The window contains a tuned block of low dielectric plastic material, such as solid PTFE, sized to minimise microwave reflection but thick enough to not distort under vacuum when heated during operation.

Condenser (8) cooled by refrigerated coolant to retain solvents and off-gases produced from the molten plastic by vacuum and heating.

Distillate Trap & Valves (9) to entrap distillate produced.

Vacuum Pump (10) to generate the vacuum to hold vessel (1) closed, lower the boiling point of the solvents in the mix and facilitate their removal and provide a non-oxidant atmosphere so that plastic and solvent were not able to ignite.

METHODS & EXAMPLES

Example 1

In order to produce a pipe for testing the properties of the combined plastic the following mix was prepared and processed with the parameters as shown in Table 5.

Table 5

Plastic Mixture shredded and granulated to	**3 – 15 mm**
PVC	20% w/w
PP	20% w/w
PET	20% w/w
HDPE	20% w/w
PAPER	4% w/w
FLY ASH	5% w/w
Carbon Black	1% w/w

Mixed Waste Solvents (Thinners)	5% w/w
XPS	5% w/w
Processing Parameters	
Production (kg)	1
Vacuum (millibar)	260
Temperature Set Point (°C)	200
Maximum Temperature (°C)	215
Ambient Temperature (°C)	26
Heating Time (mins)	45

The molten plastic was transferred from the vessel and allowed to cool in approximately 200 g mounds, in ambient air until solid and at ambient temperature. After several days the un-formed mounds were reduced to a fine, approximately 20 mesh, powder by granulation and pulverisation.

The powder was then reheated in the apparatus as previously described, and the molten plastic pressed into a mould with nominal ID of 375 mm corresponding to the dimensions of similar standard Class 2 concrete or Class 2 reinforced concrete pipe.

After cooling, this sample was subjected to functional testing by independent laboratories and the following results were obtained as shown in Table 6.

TABLE 6

TEST	RESULT
HYDROSTATIC	980 kPa
DENSITY	1.1
HARDNESS	60, 67
FLEXURAL STRENGTH	15.7 mPa
COMPRESSION	42.2 mPa
% INCREASE IN WEIGHT AFTER IMMERSION IN WATER	0.14, O.53

The pipe produced was found to have characteristics which were far better than a Class 2 concrete pipe of similar dimensions except that it was less than half the weight, expected for such a concrete pipe.

Further work on the process development was performed and it was found, somewhat surprisingly, that the process according to the present invention could be combined into a one step process, eliminating the need to waste heat and time and no longer requiring the regrind step. This has become a preferred process of the invention and will now be described in Example 2.

The choice of plastic mix is by way of example only and is not limited to the proportions or types of plastics chosen for the example.

Example 2

In order to produce a pipe for testing the properties of the combined plastic the following mix was prepared and processed with the parameters as shown in Table 7.

TABLE 7

Plastic Mixture shredded and granulated to	**1 – 5 mm**
PVC	5% w/w
PP	23% w/w
PET	18% w/w
HDPE	20% w/w
PA (NYLON)	5%
PE/GLASS MIXTURE	3%
LDPE/GLASS/ALUMINIUM/PAPER LABELS	5%
PC (POLYCARBONATE)	5%
Carbon Black	1% w/w
Mixed Waste Solvents (Thinners)	5% w/w
XPS	5% w/w
Processing Parameters	
Production (kg)	2.2

Vacuum (millibar)	270
Microwave Power (kW)	1
Microwave Frequency (gHz)	2.45
Temperature Set Point (°C)	190
Maximum Temperature (°C)	210
Ambient Temperature (°C)	21
Heating Time (mins)	50

The molten plastic was then pressed into a mould with dimensions of an 80 mm ductile iron pipe, the dimensions also corresponding to the now discontinued asbestos cement concrete pipe (OD 96 mm x ID 81 mm). The section of pipe formed and tested was 400 mm long.

After cooling, this sample was subjected to functional testing by independent laboratories and the following results were obtained as shown in Table 8.

TABLE 8

TEST	RESULT
HYDROSTATIC	1950 kPa
DENSITY	0.979, 1.031
HARDNESS	64 – 71
FLEXURAL STRENGTH	15.7 mPa
COMPRESSION	42.2 mPa
% INCREASE IN WEIGHT AFTER IMMERSION IN WATER	0.32 – 1.37

The pipe produced was found to have characteristics which exceeded Class 2 concrete pipe and compared favourably with Class 12 UPVC pressure pipe. Again pipe produced achieved these parameters while weighing less than half the weight of a corresponding concrete pipe.

Example 3

In order to assess the potential to incorporate LDPE shopping bags without compromising the product's integrity, the following mix was used to produce a pipe and processed with the parameters as shown in Table 9.

TABLE 9

Plastic Mixture shredded and granulated to	**1 – 5 mm**
PVC	5% w/w
PP	15% w/w
PET	17% w/w
HDPE	17% w/w
LDPE (PLASTIC SHOPPING BAGS)	20%
PET/PAPER LABELS/LIDS	5%
LDPE/GLASS/PAPER LABELS	5%
Carbon Black	1% w/w
Mixed Waste Solvents (Thinners)	5% w/w
XPS	5% w/w
Processing Parameters	
Production (kg)	2.2
Vacuum (millibar)	270
Microwave Power (kW)	1
Microwave Frequency (gHz)	2.45
Temperature Set Point (°C)	190
Maximum Temperature (°C)	210
Ambient Temperature (°C)	22
Heating Time (mins)	50

The molten plastic was then pressed into.a mould with dimensions as described in Example 2. The resultant pipe did not differ in outcome, appearance or functionality to that produced in Example 2, thus confirming the ability of the process to

accommodate LDPE shopping bags, without modification to the process and without compromising the product.

It will be appreciated by persons skilled in the art that numerous variations and/or modifications may be made to the invention as shown in the specific embodiments without departing from the spirit or scope of the invention as broadly described. The present embodiments are, therefore, to be considered in all respects as illustrative and not restrictive.

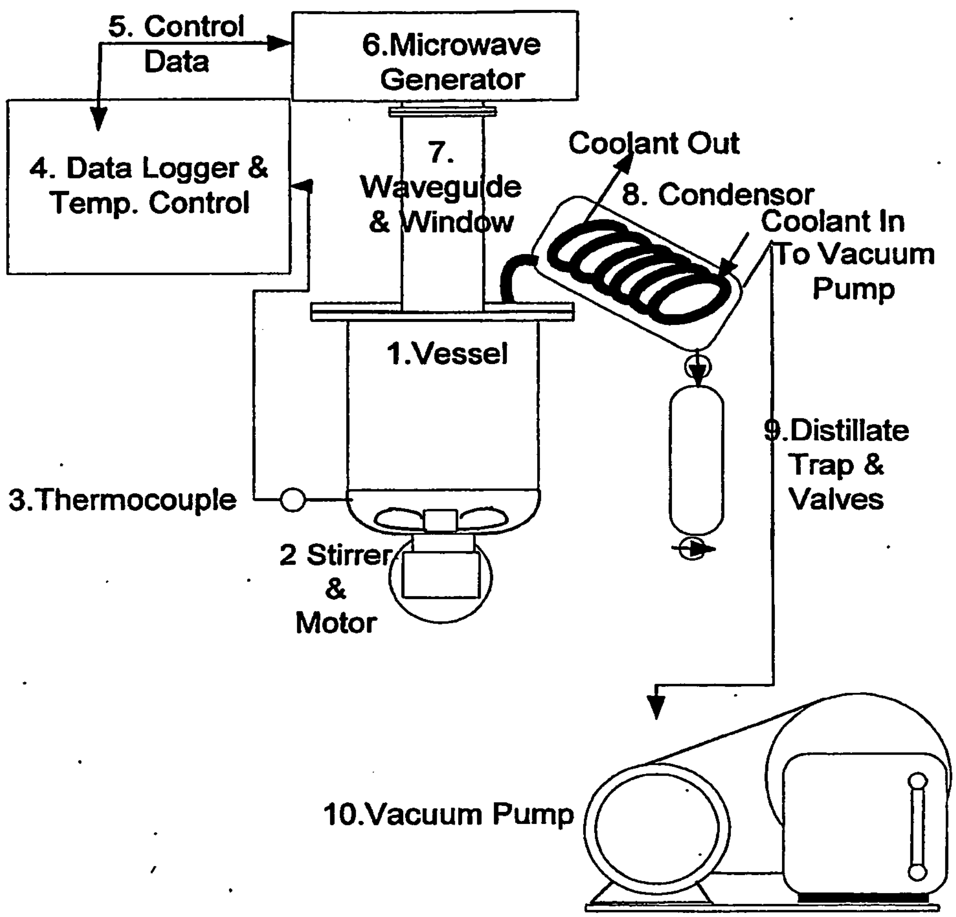
Dated this thirteenth day of October 2003

Cycloplas Holdings Pty Ltd

Patent Attorneys for the Applicant:

ALLENS ARTHUR ROBINSON
PATENT & TRADE MARKS ATTORNEYS

FIGURE 1


5. Control
Data
6.Microwave
Generator
4. Data Logger &
Temp. Control
7.
Waveguide
& Window
Coolant Out
8. Condensor
Coolant In
To Vacuum
Pump
1.Vessel
9.Distillate
Trap &
Valves
3.Thermocouple
2 Stirrer
&
Motor
10.Vacuum Pump

Document made available under the Patent Cooperation Treaty (PCT)

International application number: PCT/AU04/001389

International filing date: 12 October 2004 (12.10.2004)

Document type: Certified copy of priority document

Document details: Country/Office: AU
Number: 2003905590
Filing date: 13 October 2003 (13.10.2003)

Date of receipt at the International Bureau: 01 November 2004 (01.11.2004)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)